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14. ABSTRACT
Hollow CoS, CoO, and iron oxide particles have all been synthesized by exploiting the nanoscale Kirkendal effect. Yolk-shell type particles have been synthesized by coating an inert seed particle with a reactive metal intermediate. Anion exchange of ZnO nanoparticls through heteroepitaxial anion exchange was shown to preserve single-crystallinity and parent-particle shape. Silver nanoparticles have been shown to undergo a reversible, noncubic distortion under pressure. Diffraction peaks from Ag nanoparticles inside hollow CoO and Fe3O4 nanoshells were used to monitor their internal pressure. The mechanical properties and fracturing of hollow nanoparticles were studied with an in-situ TEM indenter. The single-particle fracturing and deformation events were correlated to their individual stress-strain maps in this way. The first-order phase transition of CdSe nanoparticles, deformation of gold spheres, and the absorption of energy by breaking hollow nanoshells have been demonstrated under shock compression.

15. SUBJECT TERMS
synthesis of nanoparticles and nano-heterostructures with complex topologies, nano-scale Kirkendall effect, heteroepitaxial anion exchange, high-pressure syncrotron X-ray diffraction, In-Situ TEM indentation, time-resolved shockwave spectroscopy

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Final Performance Report

Energy Absorbing Media Based Upon Nanocrystals Of complex Shape and Topology AFOSR Grant No. FA9550-07-1-0334 15 April 2007 – 14 April 2008

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Energy absorbing media based upon nanocrystals of complex shape and topology

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ABSTRACT

There are numerous situations in which a light weight composite with high energy absorbing capabilities can be of use in the Air Force mission, for protection against impact of projectiles or laser beams. Advances in nanoscience have created a new opportunity to design such composites, as it is now possible to blend inorganic nanocrystals of controlled geometry and topology inside plastics. We propose to investigate new types of colloidal inorganic nanocrystals of complex shape and topology, which have the potential to act as energy absorbers during a collision, or in the shock wave generated by an intense laser beam. Nanocrystals respond to mechanical deformations in ways that are completely distinct from bulk materials, and in many cases they exhibit more ideal behavior, with the ability to deform to a higher degree than the bulk material before fracture occurs. Recently we developed new methods to prepare hollow nanocrystals, nested hollow nanocrystals, branched nanocrystals, and hyper-branched nanocrystals. It is of considerable interest to try to create new composites that contain these novel colloidal nanocrystals embedded in a soft matrix. This proposal brings together three components for such a program: synthesis of new colloidal nanocrystals with potential energy absorbing properties; tests of mechanical deformation of isolated nanocrystals using a range of experimental techniques (indentation, scanning probe microscopy, hydrostatic pressure, and laser-induced shock waves); creation of composites of the nanocrystals in plastics, suitable for mechanical testing, for instance at Air Force laboratories.

The National Environmental Policy Act of 1969 (NEPA) requires Federal agencies to consider potential environmental concerns of major federal undertakings. This includes research projects funded by the Air Force Office of Scientific Research (AFOSR). Under the Air Force Environmental Impact Analysis Process, all projects must have an environmental assessment or environmental impact statement completed UNLESS they qualify for a categorical exclusion from this requirement. In order to qualify for this categorical exclusion, proposed research must be normal and routine basic or applied research confined to the laboratory and in compliance with all safety, environmental, and natural resource conservation laws. The following documentation must be completed in order to assist AFOSR in determining whether the proposed research meets the criteria for such categorical exclusion.

The University of California, Berkeley and **Prof. A. Paul Alivisatos**
(Name of Proposing Institution) (Name of Investigator)
hereby certify as follows:

1. All research to be performed under the proposal for research Energy absorbing media based upon nanocrystals of complex shape and topology
(Research Title)

will be confined to the laboratory, except as disclosed below:

2. All research identified in number 1 above, will be conducted in compliance with all safety, environmental, and natural resource conservation laws, except as disclosed below

3. The proposed research does not involve major construction or remodeling of buildings used as research or test facilities.

4, Any additional information that will assist AFOSR in accomplishing the required environmental determination:

The parties signing this certification below understand that the Air Force Office of Scientific Research will rely on the certification in making determinations under the Air Force Environmental Impact Analysis Process and whether the proposed research qualifies for a categorical exclusion.

Linda Cusentino for Susan Hedley
Senior Administrative Analyst — 2/14/06
(Signature of Authorized Official of the Institution) (Date)

(Signature of Principal Investigator) — 2/13/06
(Date)

Statement of objectives:

- Develop new energy-absorbing composites based upon colloidal nanocrystals of complex shape and topology

- Synthesis of hollow nanocrystal ceramics with controlled diameter and wall thickness
- Synthesis of nested hollow nanocrystals with controlled wall spacings
- Synthesis of branched and hyper-branched nanocrystals with varying branch patterns

- Preparation and imaging of nanocrystal-polymer composite blends

- Indentation studies of nanocrystals with an *in-situ* TEM
- Small and wide angle high pressure X-ray diffraction studies of nanocrystals
- Laser-induced shock wave studies of colloidal nanocrystals

Introduction

Many advanced composite materials with superior mechanical properties contain inorganic components dispersed inside a plastic. These may consist for instance of carbon fibers[1] or sheets of silicate or montmorillonite dispersed within a plastic[2]. In the last decade, there have been significant advances in the creation of nanoscale inorganic building blocks, with increasing control over size and shape. The mechanical properties of these new nanoparticles are unusual, in that they can be deformed to a much higher degree than the corresponding bulk solid before fracture (albeit accompanied by plastic deformation and generation of dislocations)[3]. This is illustrated in Figure 1, with a new nanoscale material produced in our laboratories, CdSe tetrapods. The figure shows tetrapods whose arms have been bent by the capillary forces of an evaporating solvent (they are on the side walls of a trench to enable visualization.) It can readily be seen that the tetrapod arms have been bent well past the angle at which the bulk solid would fracture[4]; yet in the nanoparticles, the system plastically deforms with the generation of dislocations, without undergoing fracture. This illustrates the fundamental goal of this proposal: to prepare new nanostructures, such as hollow nanocrystals and branched nanocrystals, with potentially novel mechanical properties; to investigate the mechanical properties of the individual components; and to prepare composites of the nanocrystals inside polymer films.

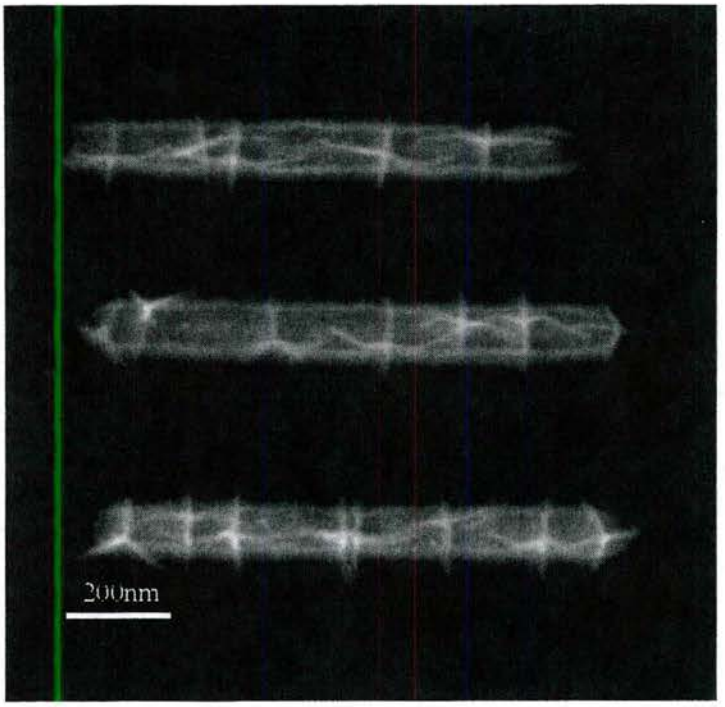


Figure 1. CdTe tetrapods deposited on the side walls of a trench by solvent evaporation. The capillary forces of evaporation have bent the arms of the tetrapods to angles well past the fracture limit of the corresponding bulk; yet on the nanocrystals this results only in plastic deformation.

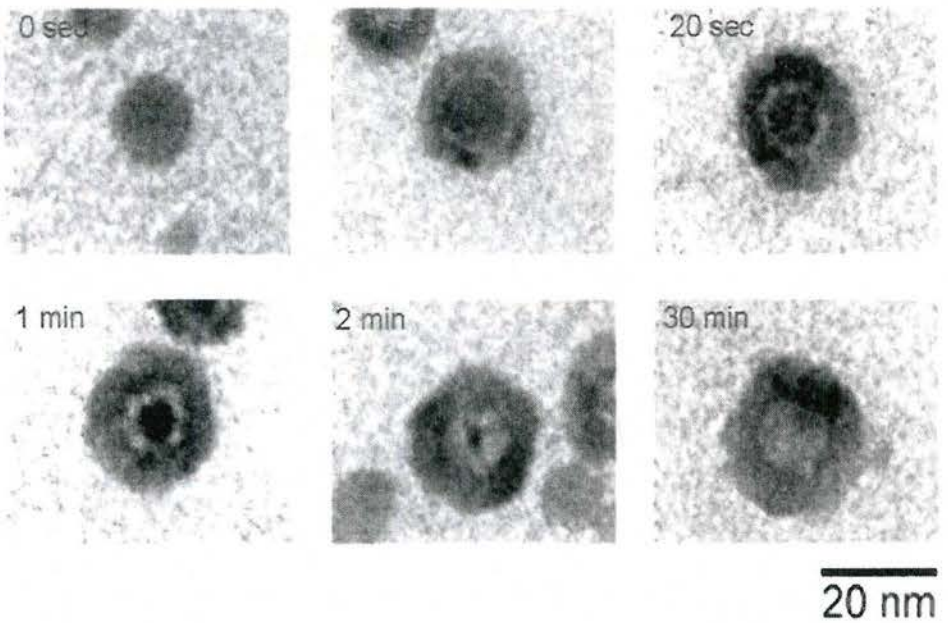


Figure 2. Sequence of events during the formation of hollow Cobalt Selenide by using the nanoscale Kirkendall effect. The total energy required to break the wall of one of these hollow nanocrystal will be an important subject of this proposal.

Synthesis of hollow ceramic nanocrystals

The AFOSR has supported work in our laboratories over the last several years, relating to the synthesis of oxide and ceramic nanocrystals. With this support, we discovered a systematic means for creating hollow nanocrystals[5] ("Formation of hollow nanocrystals through the nanoscale Kirkendall Effect," *Science* **2004,** 304, (5671), 711-714.). In this method, one starts with a solid nanocrystal, for instance cobalt or iron (although many materials can be used, for instance Zn, Cd, etc.); these nanocrystals are dispersed in solution, heated, and then exposed to a low concentration of oxygen or elemental chalcogenide (S, Se, Te); the nanocrystals are transformed into the corresponding compound (for instance CoO, or CdSe), and the resulting particles are hollow. The key concept is illustrated in Figure 2. Here we see the evolution of the formation of hollow Cobalt Selenide from the reaction of elemental Se with a solid Co nanocrystal. In the first stage of the reaction, a thin layer of Cobalt Selenide forms at the surface. Further reaction proceeds on the outermost surface, because Co ions diffuse outwards through the CoSe layer, faster than Se ions diffuse inwards. This disparity in diffusion results in vacancies being left behind. As the vacancies super-saturate, they coalesce into larger voids, which finally merge into a single central void. We have mapped out the dependence of this process on temperature and reaction rate (an example is shown in Figure 4 on the next page). The walls of the hollow nanocrystals are polycrystalline, with approximately 10 to 20% vacancy distributed within the wall (Figure 3, below).

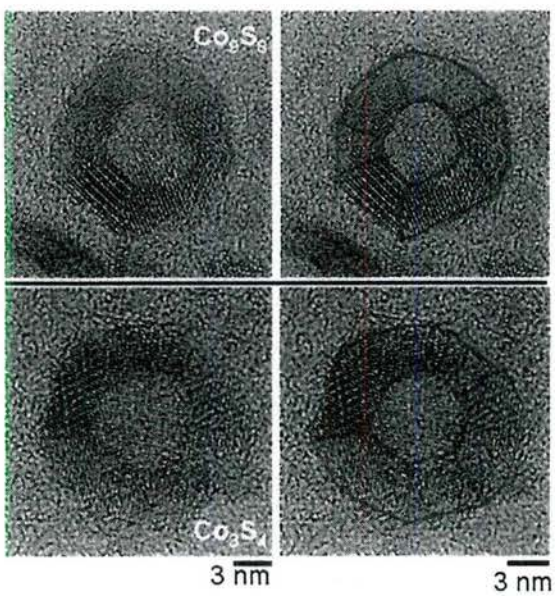


Figure 3. The hollow nanocrystals produced by the nanoscale Kirkendall effect are polycrystalline. The shells consist of grains which are oriented radially. In between the grains are boundaries and numerous vacancies. The average vacancy concentration inside the shell wall is 10-20%

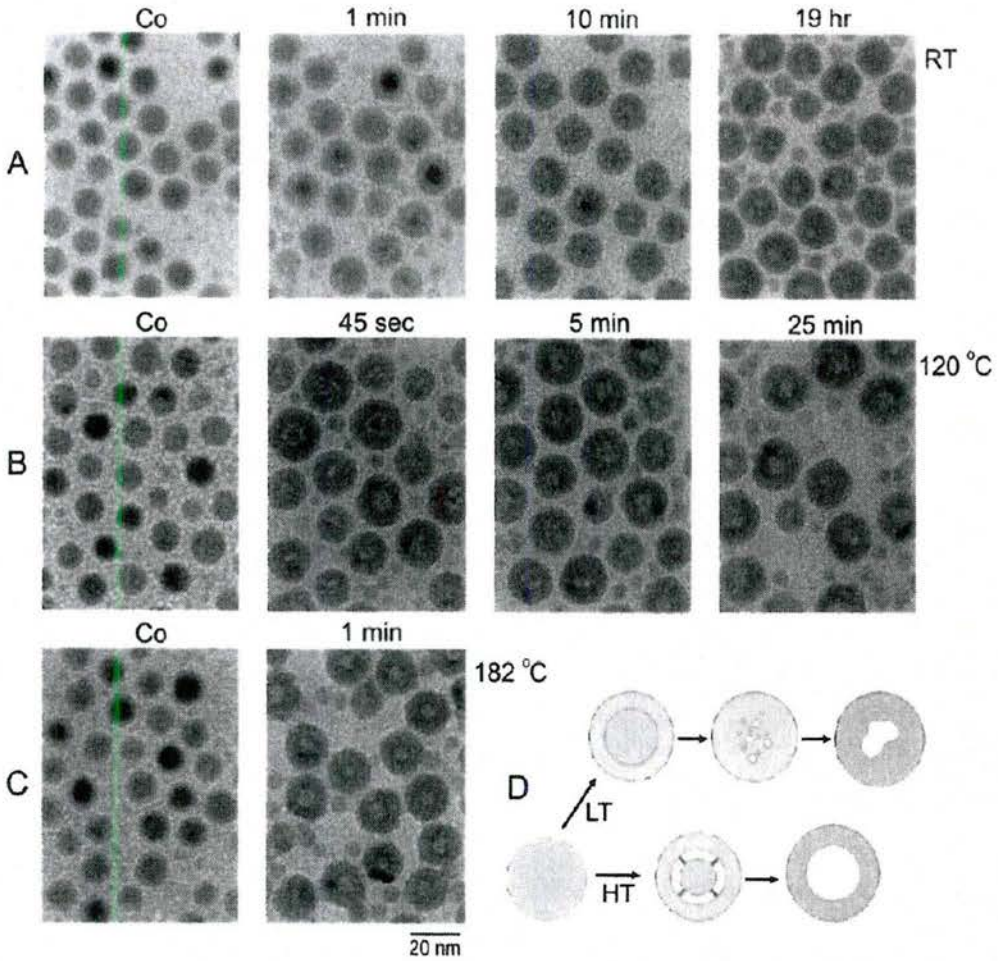


Figure 4. shows the evolution of hollow nanocrystals as a function of temperature and time (Cobalt Sulfide). At low temperature the supersaturated vacancies are not able to coalesce into a single void. At higher temperatures, a single void forms. At still higher temperatures (above 200 C, not shown here) the central void collapses, to yield a lower total surface area.

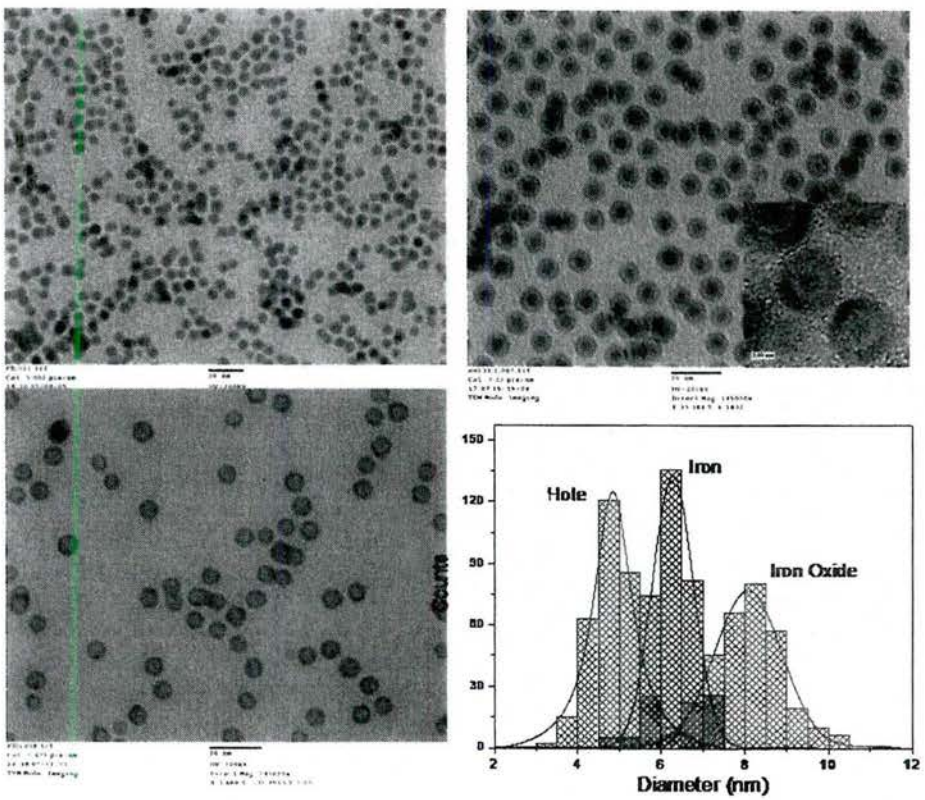


Figure 5. A wide range of materials can be made as hollow nanocrystals. This figure illustrates the formation of hollow gamma Iron Oxide (Fe2O3). The initial Iron particles are exposed to Oxygen. The central void is a bit smaller than the original Iron, demonstrating the extent to which the oxygen penetrates the initial oxide shell. Many materials can be made hollow this way.

Mechanical deformation studies of hollow nanocrystals using an *in-situ* TEM

We propose to investigate the mechanical deformation of hollow nanocrystals using indenters and atomic force microscopes. The indenter studies will be performed in close collaboration with Dr. Andy Minor of the National Center for Electron Microscopy, who has built an indenter system within a Transmission Electron Microscope. We have performed a set of preliminary studies on hollow CdS crystals of 100nm diameter, with 20nm wall thickness. These studies show that the force-displacement curve is nearly linear over a wide range of displacements, well into the regime of substantial plastic deformation of the hollow crystals (flattening). At a critical force, the hollow crystal collapses as the wall ruptures. We propose to investigate these force displacement curves as a function of diameter and wall thickness. In our preliminary studies, we have determined the threshold for wall fracture as a function of these variables. A typical force-displacement curve is shown on the next page.

We have estimated how much energy can be dissipated up to the threshold of breaking by a composite of nearly close packed particles of CdS with 100nm diameter and 20nm walls. We find that this is 5J/g. This compares to 33J/g for Kevlar and 12J/g for Carbon fiber[6]. It is important to note that no effort has yet been made to optimize this result. A main goal of the present project will be to seek an optimization of the nanocrystals for this property by variation of diameter, wall thickness, and even composition. More importantly, we will also consider nested hollow nanocrystals, which can contain a much greater density of walls per unit volume than the simple hollow particles shown thus far.

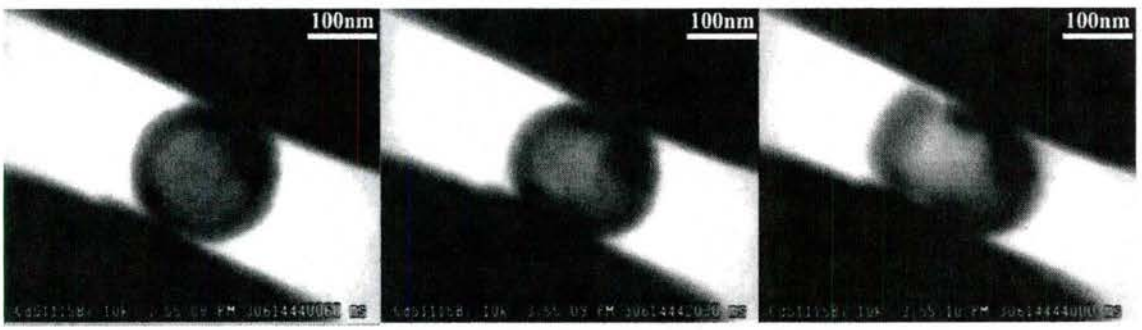


Figure 6. Snapshots from a movie showing an indenter breaking a hollow CdS particle. The experiment is performed inside a Transmission Electron Microscope. At first the particle is flattened, then it cracks open. The force vs. displacement curve is recorded along with the images.

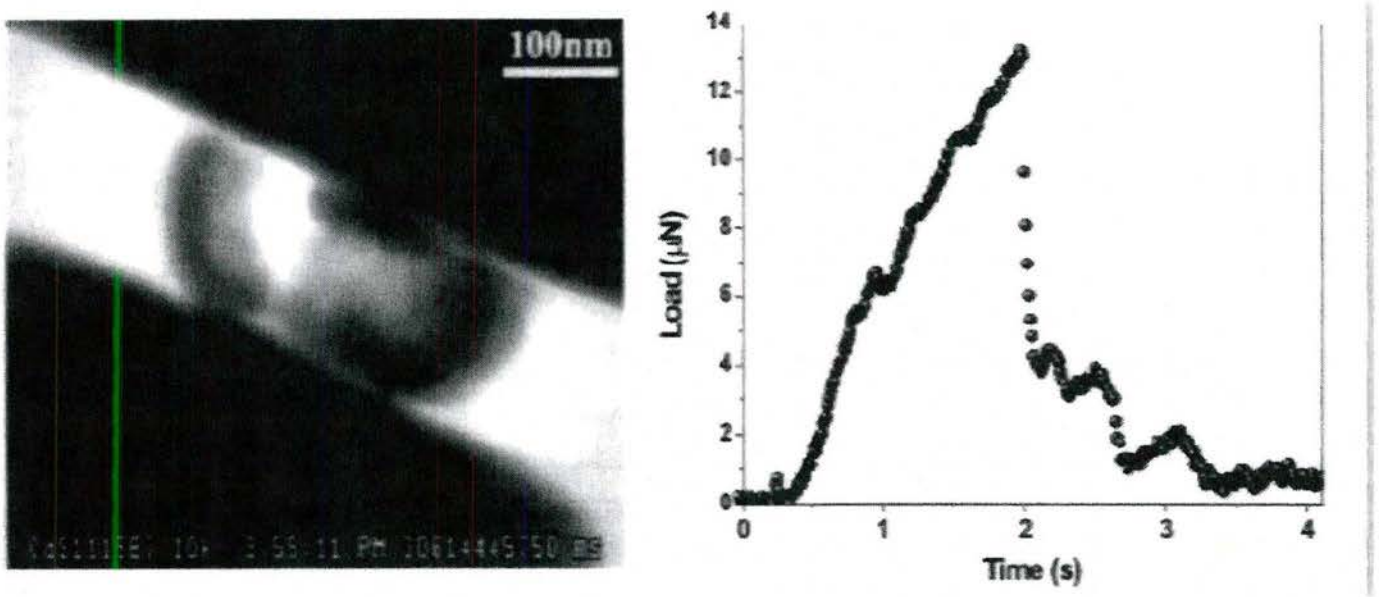


Figure 7. A force vs. displacement curve is generated along with each movie of the crushing of a hollow nanocrystal. At low displacements, the curve is nearly linear, even though once contact occurs there is some irreversible plastic deformation. The abrupt cracking of the shell is clearly visible at 2s. The integral of the force displacement curve up to this point gives us the total energy required to break a single shell.

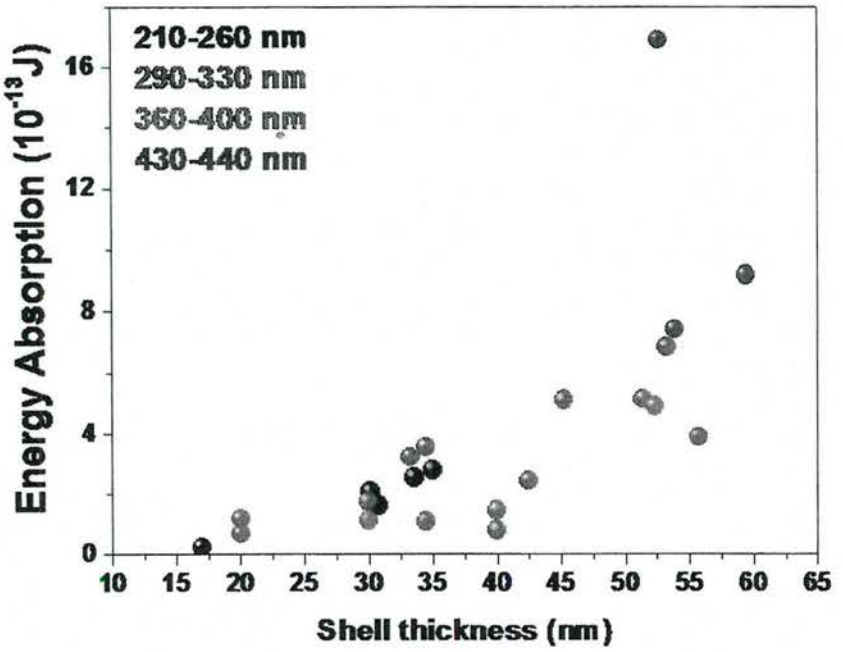


Figure 8. Total energy required to break the shell, as a function of the shell thickness, for hollow crystals of different diameters (color coded). A goal of this proposal will be to systematically study these dependencies, and to understand their basis.

High pressure studies of hollow nanocrystal collapse threshold

We propose to investigate the influence of hydrostatic high pressure on the hollow nanocrystals, with the goal in mind of finding the critical pressure at which the wall of the hollow crystal will collapse, as a function of the wall thickness and diameter. The hollow nanocrystals will be dispersed inside a soft hydrostatic pressure transmitting medium (methanol-ethanol or ethyl cyclohexane), and then pressurized in a diamond anvil cell. Small and wide angle X-ray diffraction patterns can be collected at the high pressure beam line of the Advanced Light Source Synchrotron. In preliminary experiments on hollow Cobalt Sulfide nanocrystals of 20nm outer diameter, 5 nm wall thickness, we found that the nanocrystals could be pressurized up to 20Gpa, without the walls collapsing. However, we did observe that the walls abruptly compacted at critical pressure of about 4 GPa. This compaction results when the softer, inter-granular regions of the hollow crystal walls are abruptly compacted. This is an irreversible process. Future studies will involve indentation on these compacted particles, as well as studies at higher pressures, aimed at finding the threshold for wall collapse.

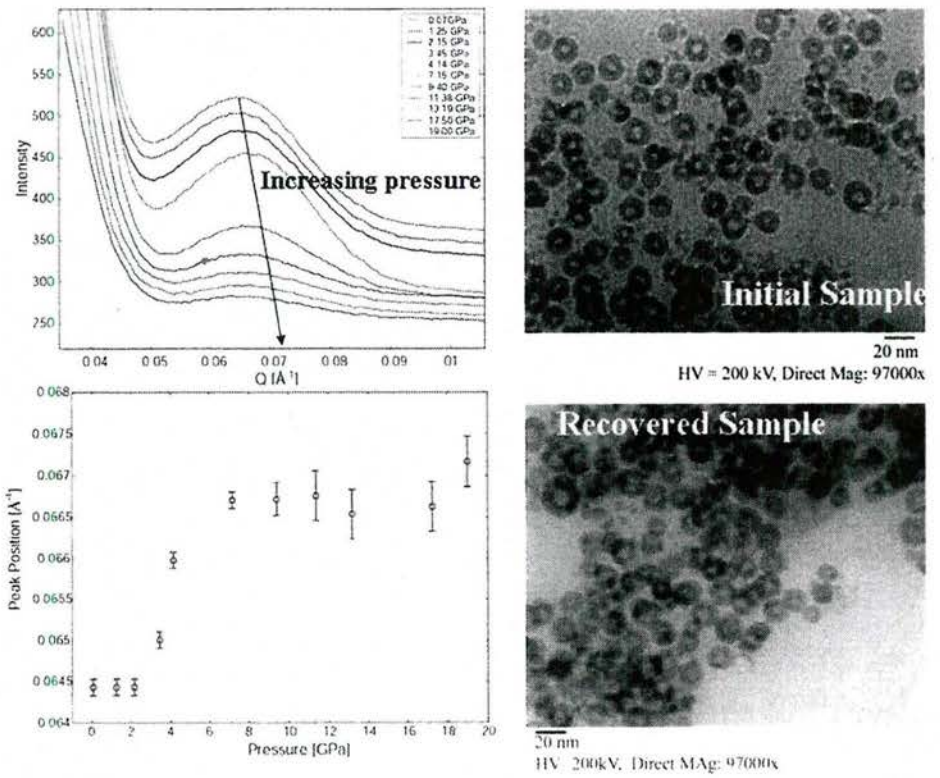


Figure 9. Small Angle X-ray Scattering as a function of hydrostatic pressure for hollow cobalt sulfide nanocrystals of 20 nm diameter and 5 nm wall thickness. The walls contract at about 4 GPa, but do not collapse all the way up to 20 GPa.

Nested hollow nanocrystals

The most straightforward way in which to increase the energy absorbing capacity per unit volume of the hollow nanocrystal based composites is to increase the number of walls, without decreasing their thickness, and maintaining a close to optimal curvature (we don't yet know the optimal curvature, this will emerge from the indentation studies of regular hollow nanocrystals). Nested hollow structures provide a natural avenue towards more energy absorbing materials. We have been investigating such approaches, and we can now prepare nested structures in a variety of ways, two of which are shown below. We will investigate the mechanical deformation of these nested hollow structures with the indenter, the AFM, and with hydrostatic high pressure techniques as part of this proposal.

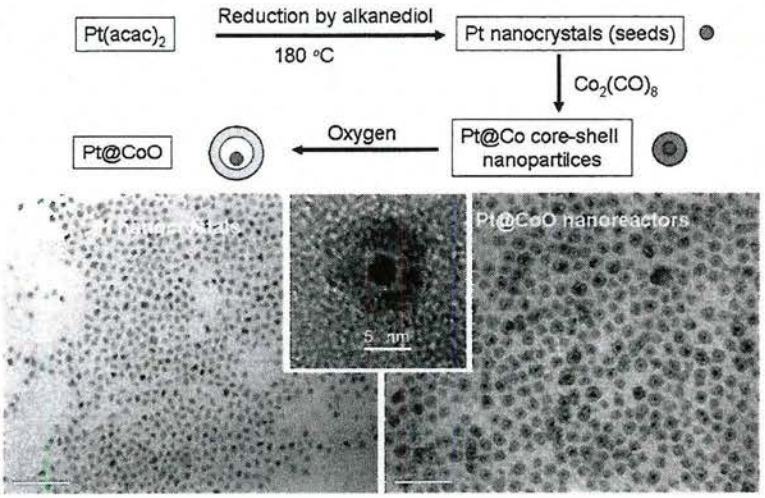


Figure 10. Scheme one for generating nested structures. An original particle, in this case Platinum, is overcoated with Cobalt. When the Cobalt is Oxidized, it becomes hollow, with a Platinum nanocrystal nested within. It is possible to subsequently react the interior component with another gas, to yield a double hollow crystal.

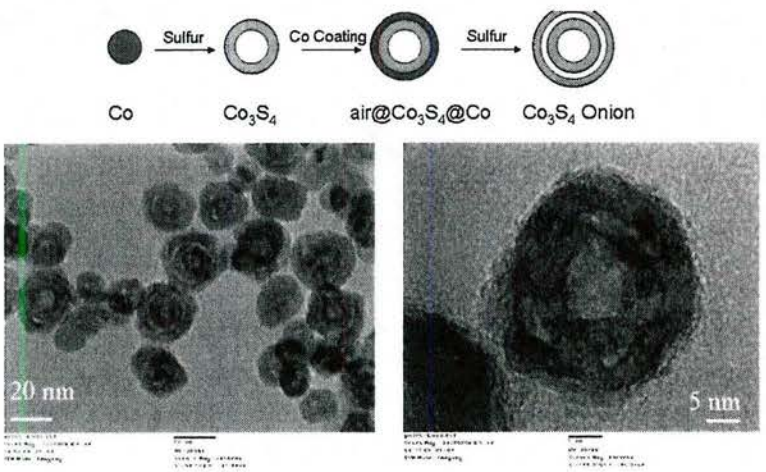


Figure 11. Scheme 2 for generating nested structures. A cobalt nanocrystal is reacted first with sulfur to create a hollow cobalt sulfide. Then this nanocrystal is overlaid. with Co. A second sulfidation step now generates nested hollow structures. As far as we can tell, this could be repeated several times, yielding a very dense network of walls

Synthesis of branched and hyper-branched nanocrystals

A very different type of topology we are investigating are branched nanocrystals, such as tetrapods, multiply branched, and hyper-branched nanocrystals. In the course of investigating the kinetics of the growth of semiconductor nanocrystals, we accidentally discovered the way to create branch points in semiconductor nanorods[7]. The idea relies upon the fact that the II-VI semiconductors have two possible bonding geometries that are closely related: zincblende, corresponding to ABCABC stacking, and Wurtzite corresponding to ABAB stacking. The zincblende stacking arrangement is favored kinetically, while the Wurtzite is favored thermodynamically. Thus by changing the growth rate it is possible to switch between the two. A small piece of zinblende can serve as branch point, since the high symmetry cubic structure naturally forms pyramidally shaped nanocrystals. These tetrahedral nanocrystals can send out hexagonally packed arms at the tetrahedral angle. We have succeeded in creating tetrapods of controlled length and diameter, as well as inorganic dendrimers of generation 2[8], and most recently, hyper-branched nanocrystals with multiple branch points[9]. In the hyper-branched structures, the branches are formed very quickly, leading to the formation of twins, which results in forward and reverse branching. The mechanical properties of these branched structures will be the subject of our investigations.

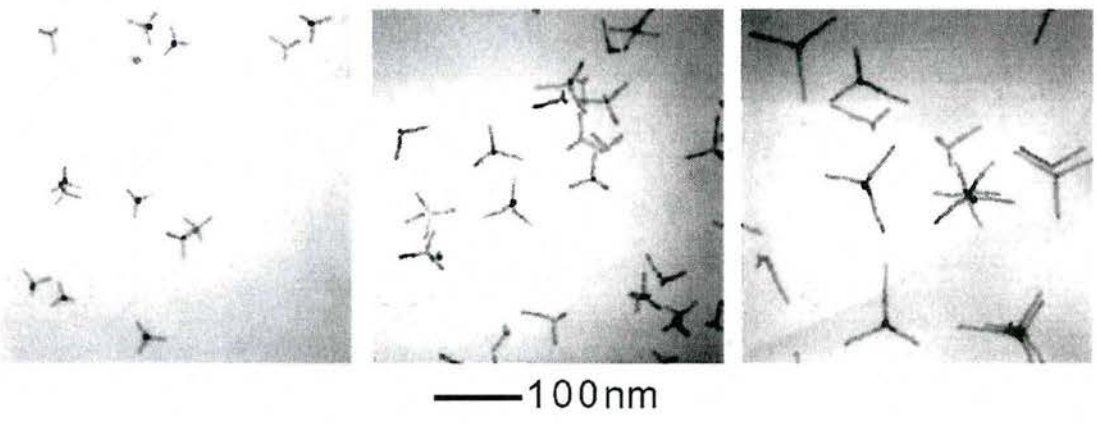


Figure 12. Controlled synthesis of branched tetrapods of CdTe, with varying arm length. The arm diameters can also be controlled.

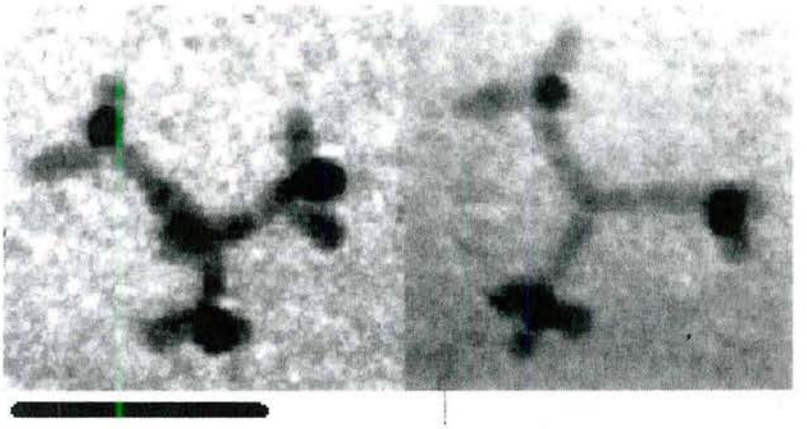


Figure 13. Generation 2 inorganic dendrimers consisting of a first generation CdTe and a second generation CdSe. The scale bar is 50 nm.

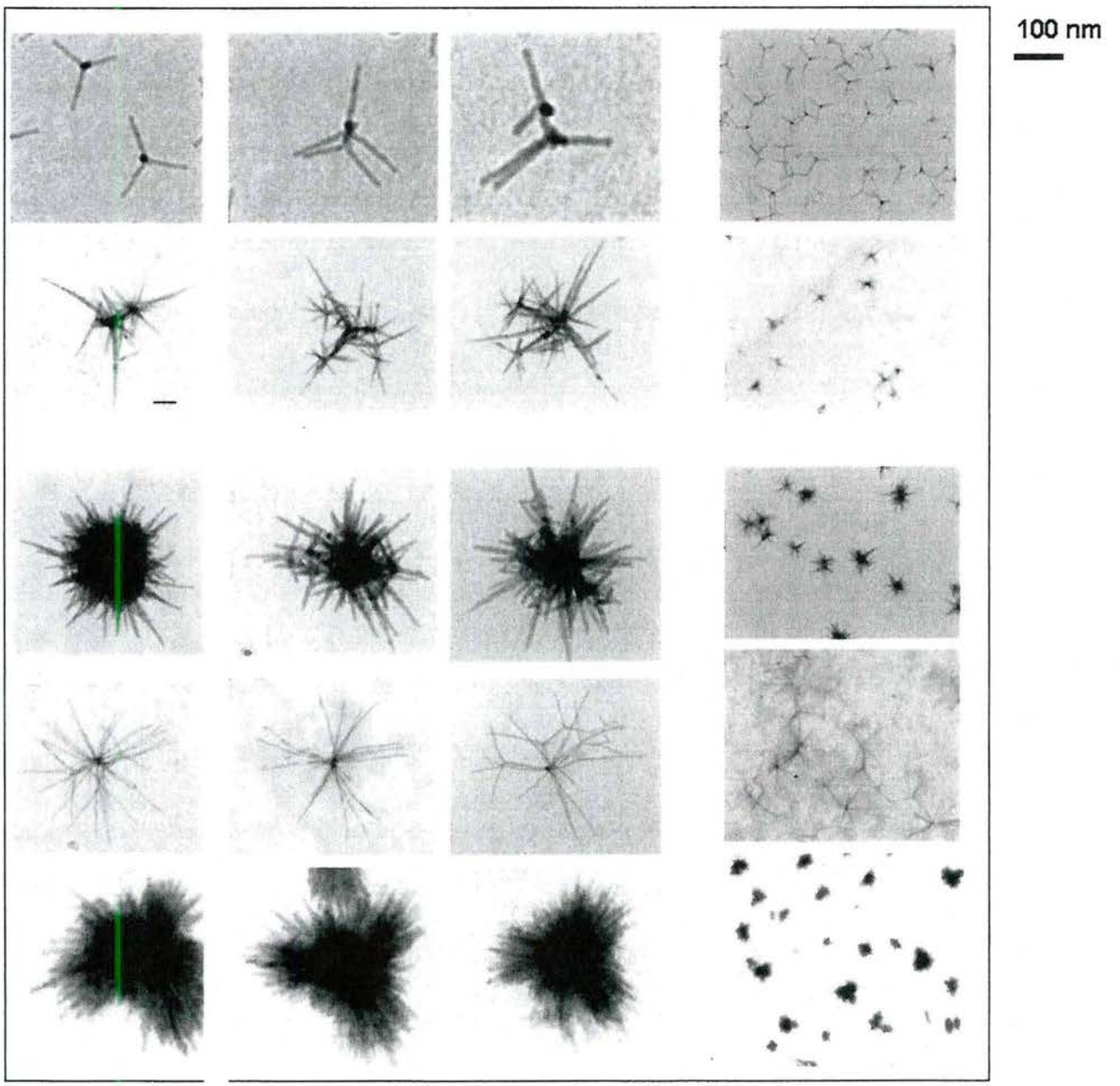
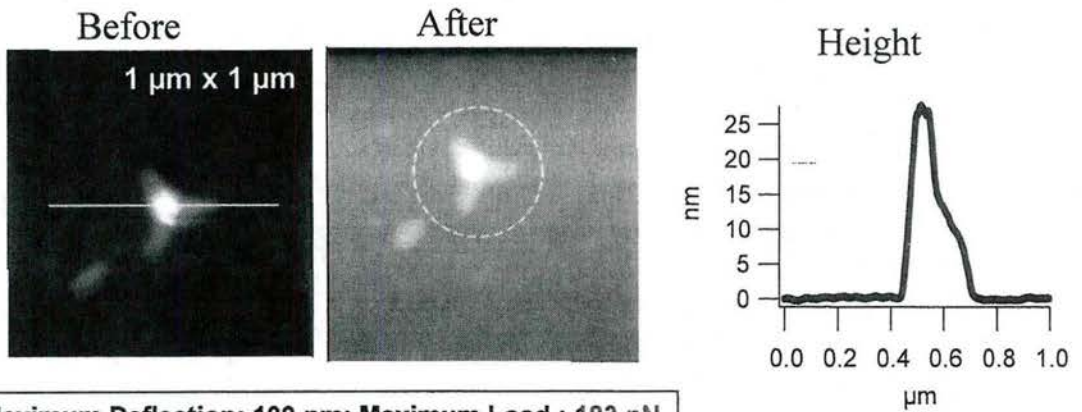


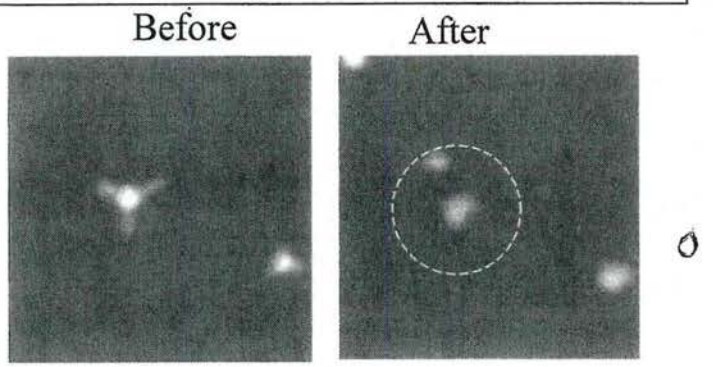
Figure 14. Hyper-branched nanocrystals of CdTe and CdSe, formed during controlled colloidal nanocrystal growth. The density and pattern of branches can be finely adjusted, to produce a distinct set of patterns.

Mechanical deformation studies of individual tetrapods – AFM and indenter

Maximum Deflection: 40 nm; Maximum Load : 73.2 nN



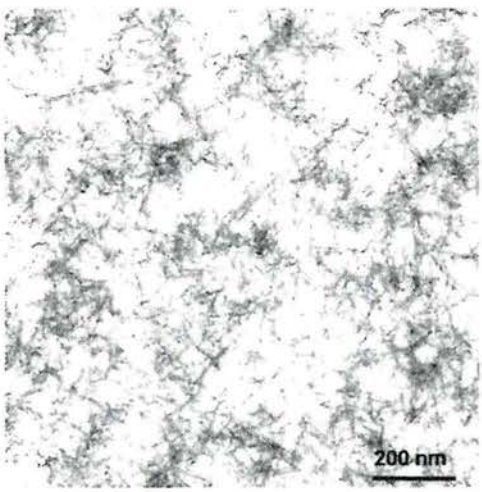
Maximum Deflection: 100 nm; Maximum Load : 183 nN



We propose to investigate the deformation limits for individual tetrapods as a function of arm length and width, using the atomic force microscope in our laboratory, as well as the *in-situ* indenter/TEM combination used to study the hollow nanocrystals. Again, we will collaborate as necessary with Dr. Andy Minor of the NCEM, and with Dr. Miquel Salmeron, the director of the imaging facility at the Molecular Foundry. In preliminary studies shown above, we were able to deform individual tetrapods. Modest forces produced only elastic deformation for a displacement of up to 40 nm; above a critical threshold, we found that the tetrapods deformed permanently. As part of this proposal, we will measure that threshold systematically for many tetrapods of controlled arm length and width.

Formation of composite structures

The studies described this far involved the investigation of the mechanical deformation of individual hollow nanocrystals and tetrapods. We believe this is the critical point at which to start these explorations. However, in order to progress towards creating a complete material system for energy absorption, we will also develop composites of these nanostructures within polymer films and polymer blocks. We have extensive prior experience loading colloidal nanocrystals into polymers (for instance for hybrid solar cells)[10], and we can specifically engineer the surfactant molecules on the surfaces of the nanocrystals in such a way as to control the dispersion of the nanoparticles. The particles will be dispersed inside the polymers for instance by spin casting from a mixture of solvents, one optimal for nanocrystals, and one for the polymer. Some of the more complex structures we have prepared, for instance the hyper-branched nanocrystals, will best be studied in the composite films shown here, rather than as individual crystals. These samples will be used in the shock wave studies, described next.



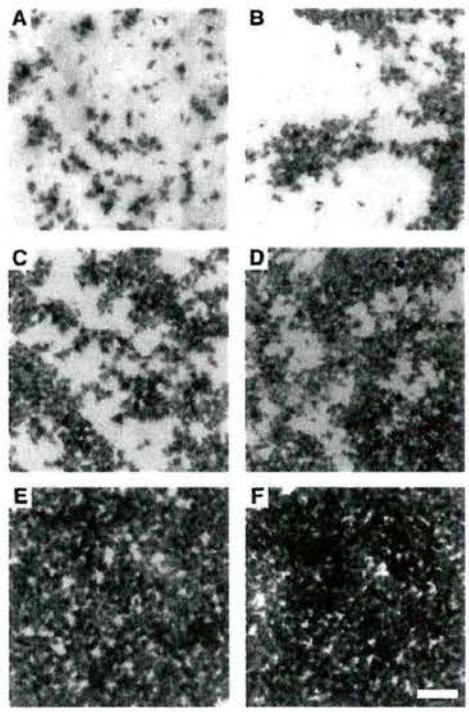


Figure 16. Above: Dispersion of nanorods in a P3Ht polymer film. Right, Controlled dispersion of hyperbranched CdSe in P3HT Polymer.

Future investigations using laser-induced shock waves

As part of a separate activity for studying solid-solid phase transitions in nanocrystals[11], we have been developing a laser-induced shock wave system (all the components are purchased and built, and the system is presently being assembled). This system is modeled on the one developed by Dana Dlott at the University of Illinois[12]. In this approach a femtosecond laser pulse is incident on a multilayer film. The film consists of an outermost aluminum film of several microns thickness, on which is a layer of pure polymer, and then a layer of composite containing the nanocrystals. The laser is incident from the back, and ablates part of the Aluminum film. This generates a shock wave that may have a transient combined pressure of 10 GPa and temperature of 1000C, for a period of a few hundred picoseconds. When completed later this year, we will use this system to shock the hollow nanocrystals and the branched nanocrystals. We will recover the samples after they have been shocked, and examine them by Transmission Electron Microscopy.

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